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Indian Standard
SPECIFICATION FOR ALUMINIUM PHOSPHIDE FORMULATIONS
(*First Revision*)

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 1980

Indian Standard

SPECIFICATION FOR ALUMINIUM PHOSPHIDE FORMULATIONS

(First Revision)

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AMENDMENT NO. 1 NOVEMBER 1987

TO

IS : 6438 - 1980 SPECIFICATION FOR ALUMINIUM PHOSPHIDE FORMULATIONS

(First Revision)

(*Page* 5, *clause* **4.1** *with Note*) — Substitute the following for the existing clause:

'**4.1** Representative samples of the material shall be drawn as prescribed in Appendix C.'

(*Page* 8, *Appendix* B) — Add the following new appendix after Appendix B:

"APPENDIX C

(*Clause* 4.1)

SAMPLING AND CRITERIA FOR CONFORMITY

C-1. GENERAL REQUIREMENTS

C-1.0 In drawing, preparing, storing and handling the test samples, the following precautions and directions shall be observed.

C-1.1 Samples shall not be taken in an exposed place.

C-1.2 Proper precautions shall be taken while drawing the samples as the material is poisonous.

C-1.3 Precautions shall be taken to protect the samples, the material being sampled and the containers from adventitous contamination.

C-2. SCALE OF SAMPLING

C-2.1 Lot — All the material in a consignment drawn from the same batch of manufacture shall constitute a lot. If the consignment is declared or known to consist of different batches of manufacture, the tubes belonging to the same batch shall be grouped together and each such group shall constitute a lot.

C-2.1.1 The samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification.

C-2.2 The number of tubes to be selected from the lot shall be according to col 1 and 2 of Table 1. When the containers containing tubes are presented in wooden cases, 10 percent of the cases shall be opened with a minimum of two cases. From the cases which are opened, 10 percent of containers shall be taken out at random. From each of the selected containers approximately equal number of tubes shall be selected to give the sample size given in col 2 of Table 1.

TABLE 1 NUMBER OF TUBES TO BE SELECTED FOR SAMPLING

LOT SIZE	NO. OF TUBES TO BE SELECTED
(1)	(2)
101 to 500	9
501 ,, 1 000	12
1 001 ,, 3 000	15
3 001 ,, 10 000	21
10 001 and above	30

NOTE — Up to 100, the sample size may be as agreed to between the purchaser and the supplier.

C 2.2.1 These tubes shall be selected at random from the lot. In order to ensure the randomness of selection, procedures given in IS : 4905-1968 'Methods for random sampling' may be followed.

C-3. TEST SAMPLE AND REFEREE SAMPLE

C-3.1 The tubes selected as in **C-2.2** shall be divided at random into three equal sets and labelled with all the particulars of sampling. One of these sets of tubes shall be for the purchaser, another for the supplier and the third for the referee.

C-3.2 Referee Sample — The referee sample shall consist of a set of sample tubes and shall bear the seals of the purchaser and the supplier (or their representatives) and shall be kept at a place agreed to between the two.

C-4. NUMBER OF TESTS

C-4.1 The number of tests mentioned in the standard shall be first conducted on the test samples marked for the purchaser. In contingencies, such as loss, spoilage, etc, of the purchaser's test samples or if the supplier so desires, the test sample marked for the supplier may be tested. In case of dispute the referee test samples shall be tested and the test results obtained from the referee test sample shall be considered as final.

C-5. CRITERIA FOR CONFORMITY

C-5.1 The lot shall be declared as conforming to the requirements of this specification if **C-5.1.1** and **C-5.1.2** are satisfied.

C-5.1.1 For requirements given in **2.2.1** and **2.3.2** tested on individual samples, each test sample satisfies the corresponding requirements.

C-5.1.2 For aluminium phosphide content given in **2.3.1**, the mean and range of the corresponding test results shall be calculated as below:

$$\text{Mean } (\bar{X}) = \frac{\text{The sum of the test results}}{\text{Number of test results}}$$

Range (R) = The difference between the highest and the lowest value obtained for the test results

The lot shall be deemed to have met the requirements given in **2.3.1** of the specification if the expression $\bar{X} = 0 \cdot 6\, R$ is greater than or equal to the minimum requirement given in this specification."

(AFCDC 6)

Printed at New India Printing Press, Khurja, India

AMENDMENT NO. 2 MARCH 1989
TO
IS : 6438 - 1980 SPECIFICATION FOR ALUMINIUM PHOSPHIDE FORMULATIONS

(First Revision)

(*Page* 4, *clause* **2.2.1**) — Substitute the following for the existing clause:

'**2.2.1** *Description* — When unpacked, the material shall be in the form of whole tablets or pellets, greyish in colour. The material shall show no evidence of spontaneous combustion when exposed to air. The weight of individual tablet shall not differ from the declared nominal value by more than the percent tolerance limits indicated below:

Nominal Weight in g	*Tolerance, percent*
As declared	±5 of the nominal weight'

(*Page* 3 *of Amendment No.* 1, *clause* C-5.1.2) — Substitute '$\overline{X} - 0{\cdot}6\,R$' *for* '$\overline{X} = 0{\cdot}6\,R$'.

(AFCDC 6)

Printed at Printwell Printers, Delhi, India

AMENDMENT NO. 3 MAY 1996
TO
IS 6438 : 1980 SPECIFICATION FOR ALUMINIUM PHOSPHIDE FORMULATIONS

(First Revision)

[*Page* 4, *clause* 2.2.1 (*see also Amendment No.* 2)] — Substitute the following for the existing:

'2.2.1 *Description* — When unpacked, the material shall be in the form of whole tablets or pellets, greyish in colour. The material shall show no evidence of spontaneous combustion when exposed to air. The average mass of 20 tablets or pellets shall not be less than the declared nominal value.'

(*Page* 4, *clause* 3.1) — Substitute 'IS 8190 (Part 4) : 1988†' *for* 'IS : 8190 (Part IV) -1979†'.

(*Page* 4, *foot-note marked* '†') — Add '(*first revision*)' at the end of text.

(*Page* 5, *clause* 5.2) — Substitute 'IS 1070 : 1992*' *for* 'IS : 1070 - 1977*'.

(*Page* 5, *foot-note marked* '*') — Substitute 'Reagent grade water (*third revision*)' for the existing title.

[*Page* 8, *Appendix* C (*inserted vide Amendment No.* 1)] — Substitute the following for the existing:

"APPENDIX C
(*Clause* 4.1)

SAMPLING PLAN FOR ALUMINIUM PHOSPHIDE FORMULATIONS

C-1 GENERAL REQUIREMENTS

C-1.0 In drawing, preparing, storing and handling the test samples, the following precautions shall be taken.

C-1.1 Samples shall not be taken in an exposed place.

C-1.2 Proper precautions shall be taken while drawing the samples as the material is poisonous.

C-1.3 Precautions shall be taken to protect the samples, the material being sampled and the containers from adventitious contamination.

C-2 SCALE OF SAMPLING

C-2.1 **Lot** —All the material in a consignment drawn from the same batch of manufacture shall constitute a lot. If the consignment is declared or known to consist of different batches of manufacture, the tubes, cans and aluminium bottles belonging to the same batch shall be grouped together and each such group shall constitute a lot.

C-2.1.1 The samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification.

C-2.2 The number of tubes, cans and aluminium bottles to be selected from the lot shall be according to columns 1 and 2 of Table 1. When the containers containing tubes, cans and aluminium bottles are presented in wooden cases, 10 percent of the cases shall be opened with a minimum of two cases. From the cases which are opened, 10 percent of containers shall be taken out at random. From each of the selected containers approximately equal number of tubes, cans and aluminium bottles shall be selected to give the sample size given in column 2 of Table 1.

Table 1 Number of Tubes, Cans and Aluminium Bottles to be Selected for Sampling

Lot Size (1)	No. of Tubes, Cans and Aluminium Bottles to be Selected (2)
101 to 500	9
501 to 1 000	12
1 001 to 3 000	15
3 001 to 10 000	21
10 001 and above	30

NOTE — Up to 100, the sample size may be as agreed to between the purchaser and the supplier.

C-2.2.1 These tubes, cans and aluminium bottles shall be selected at random from the lot. In order to ensure the randomness of selection, procedures given in IS 4905 : 1968 'Methods for random sampling' may be followed.

C-3 TEST SAMPLE AND REFEREE SAMPLE

C-3.1 The tubes, cans and aluminium bottles selected as in **C-2.2** shall be divided at random into three equal sets and labelled with all the particulars of sampling. One of these sets of tubes, cans and aluminium bottles shall be for the purchaser, another for the supplier and the third for the referee.

C-3.2 Referee Sample — The referee sample shall consist of a set of sample tubes, cans and aluminium bottles marked for this purpose and shall bear the seals of the purchaser and the supplier (or their representatives) and shall be kept at a place agreed to between the two so as to be used in case of a dispute.

C-3.3 For the purpose of requirement given in **2.2.1** of the specification, 20 tablets/pallets shall be drawn from the test samples. These tablets/pallets shall be selected at random from the chosen tubes/cans/aluminium bottles covering the top, middle and bottom layers of the each package.

C-4 CRITERIA FOR CONFORMITY

C-4.1 The lot shall be declared as conforming to the requirements of this specification if **C-4.1.1**, **C-4.1.2** and **C-4.1.3** are satisfied.

C-4.1.1 For requirement given in **2.2.1** individual test results shall satisfy the corresponding requirements.

C-4.1.2 For requirements given in **2.3.2** individual test results shall satisfy the corresponding requirements.

C-4.1.3 For aluminium phosphide content given in **2.3.1** the mean and range of the corresponding test results shall be calculated as below:

$$\text{Mean } (\bar{X}) = \frac{\text{The sum of the test results}}{\text{Number of test results}}$$

Range (R) = The difference between the highest and the lowest value obtained for the test results

The lot shall be deemed to have met the requirements given in **2.3.1** of the specification if the expression $\bar{X} - 0.6\ R$ is greater than or equal to the minimum requirement given in this specification.''

(FAD 1)

Indian Standard

SPECIFICATION FOR ALUMINIUM PHOSPHIDE FORMULATIONS

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 25 August 1980, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Aluminium phosphide in the form of tablets and pellets has lately become popular especially in fumigating stored foodgrains for protection against storage pests. Its popularity is mainly due to the convenience of its application and efficacy in comparison to other conventional fumigants. This fumigant has also been found useful in the control of rodents living in burrows. This formulation in contact with environmental moisture, releases 33 percent of its mass as phosphine gas.

0.3 While formulating this standard, the committee, responsible for the preparation of this standard, discussed at length the need for a warning gas in aluminium phosphide formulations, separate from phosphine, which itself serves the purpose. It has been established that aluminium phosphide tablets pellets without a separate warning gas can be used successfully without causing any untoward incident. It is more so when its use has been restricted to experienced pest control operators.

0.4 This standard was published in 1972. This revision incorporates the amendment issued to earlier version of the standard and also the modified test method to detect the initial evolution of phosphine gas.

0.5 In the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968, and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for aluminium phosphide formulations in the form of tablets/pellets.

2. REQUIREMENTS

2.1 The material shall essentially consist of:

a) aluminium phosphide as the only active ingredient,

b) a chemical fire retardant, and

c) a suitable binding material.

2.1.1 If required by the purchaser, the tablets or pellets may also contain chemicals emanating a separate innocuous warning gas, such as, ammonia (*see* **0.3**)

2.2 Physical

2.2.1 *Description* — When unpacked, the material shall be in the form of whole tablets or pellets, greyish in colour. The material shall show no evidence of spontaneous combustion when exposed to air.

2.3 Chemical

2.3.1 *Aluminium Phosphide Content* — When determined by the method prescribed in Appendix A, aluminium phosphide content shall be not less than 56 percent by mass.

2.3.2 The tablets/pellets shall not evolve any gas as observed by disintegration of tablets/pellets (losing their original shape) up to 30 minutes of their unpacking from the original container and after subjecting them to the test described in Appendix B.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to the requirements given in IS : 8190 (Part IV)-1979†.

---

*Rules for rounding off numerical values (*revised*).

†Requirements for packing of pesticides: Part IV Fumigants.

3.2 Marking — The container shall bear legibly and indelibly the following information, and other labelling provisions required under the Insecticides Act and Rules:

a) Name of the material;

b) Name of the manufacturer;

c) Date of manufacture;

d) Batch number;

e) Net mass of contents;

f) Aluminium phosphide content, percent (*m/m*); and

g) The minimum cautionary notice as worded in the Insecticides Act and Rules.

3.2.1 The container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 'Indian Standard methods for sampling of pesticides and their formulations' (*under preparation*).

NOTE — Till such time the standard under preparation is published, the samples shall be drawn as agreed to between the concerned parties.

5. TESTS

5.1 Tests shall be carried out by the appropriate methods referred to in **2.3.1** and **2.3.2**.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Specification for water for general laboratory use (*second revision*).

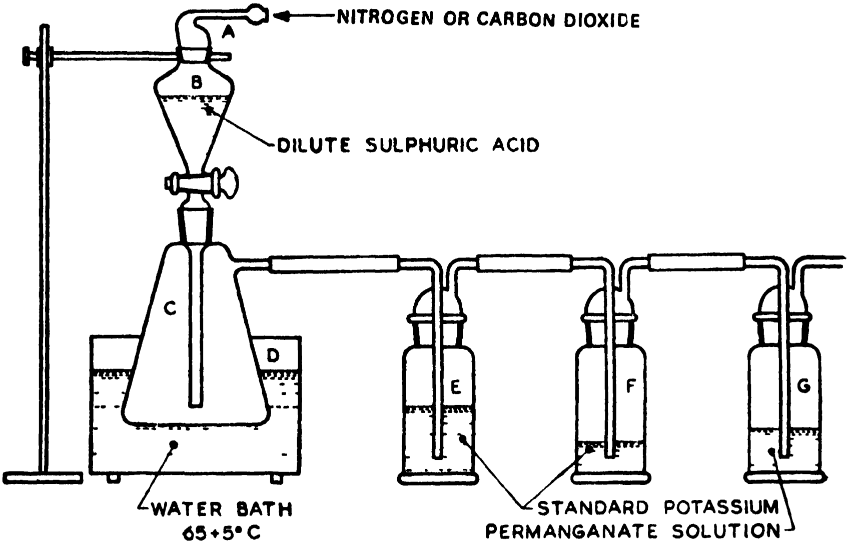
APPENDIX A

(*Clause* 2.3.1)

DETERMINATION OF ALUMINIUM PHOSPHIDE CONTENT

A-1. APPARATUS

A-1.1 The assembly of the apparatus is shown in Fig. 1.


NITROGEN OR CARBON DIOXIDE
A
B
DILUTE SULPHURIC ACID
C
D
E
F
G
WATER BATH
65+5°C
STANDARD POTASSIUM PERMANGANATE SOLUTION

A = Adapter tubing (for nitrogen gas)
B = Separating funnel conical shape with cone and socket 250 ml
C = Reaction flask (Buchner) 250 ml
D = Thermostatically controlled water-bath
E =
F = } Absorption bottles (gas wash bottles) (200/250 ml each)
G =

FIG. 1 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF ALUMINIUM PHOSPHIDE

A-1.2 The apparatus consists of a 250-ml reaction flask *C* with a standard interchangeable socket. A 250-ml separating funnel *B* and delivery tube (PVC tube surgical soft type) are to be connected according to Fig. 1. The side tube is serially connected with three 200/250 ml absorption bottles *E*, *F* and *G* respectively. To the mouth of the separating funnel is attached a nitrogen or carbon dioxide inlet tube *A* as indicated in Fig. 1. The reaction flask is so mounted on a stand where it is possible to immerse it in the water bath *D* maintained at a temperature of 65 ± 5°C.

A-2. REAGENTS

A-2.1 Standard Potassium Permanganate Solution — approximately 0·5 N.

A-2.2 Dilute Sulphuric Acid — 10 percent (*m*/*v*) and 1 : 1 (*v*/*v*).

A-2.3 Nitrogen Gas or Carbondioxide Gas — from a cylinder.

A-2.4 Standard Oxalic Acid Solution — approximately 0·5 N and acidified with sulphuric acid. Weigh accurately about 15·8 g of oxalic acid ($H_2C_2O_4 \cdot 2H_2O$) and dissolve in about 200 ml of water contained in a 500-ml volumetric flask. Add to the volumetric flask 125 ml of sulphuric acid [1 : 1 (*v*/*v*)], make up the volume with water and mix.

A-3. PROCEDURE

A-3.1 Measure 100 ml of standard potassium permanganate solution into the gas wash bottles (absorption bottles) *E*, *F* and *G*. Assemble the apparatus as shown in Fig. 1 (without dilute sulphuric acid in the separating funnel *B*). Pass the nitrogen or carbon dioxide gas slowly through the apparatus so as to displace the air. Weigh accurately about 0·2 to 0·3 g of the material and transfer it into the reaction flask *C* quantitatively.

A-3.2 Disconnect the nitrogen gas or carbon dioxide gas tube and place 100 ml of dilute sulphuric acid in the separating funnel *B*. Connect the nitrogen or carbon dioxide gas tube to the funnel again. Add the dilute sulphuric acid to the reaction flask slowly, regulating the rate of addition in such a way that a steady stream of bubbles appears in the absorption bottles *E*, *F* and *G*. When the addition of dilute sulphuric acid is complete, adjust the pressure of nitrogen or CO_2 gas so that a steady flow of bubbles is maintained in the reaction flask and the absorption bottles *E*, *F* and *G*. During this process, immerse the reaction flask *C* in the water bath maintained at a temperature of 65 ± 5°C. Continue the reaction for at least one hour. Sweep the last traces of phosphine from the flask with more rapid stream of nitrogen or carbon dioxide for at least 5 minutes. At the end of the reaction and sweeping period, disconnect the apparatus and quantitatively transfer the reduced potassium permanganate solution, contained in the three absorption bottles E, F and G to a 1 000-ml or a convenient size beaker. Rinse the absorption bottles and the connecting tubes with 100 ml of standard oxalic acid solution, taking care to dissolve all the manganese dioxide. Add the rinsings to the reduced potassium permanganate solution contained in the beaker. Rinse the absorption bottles and connecting tubes with water and transfer the rinsings to the same beaker. Warm the contents of the beaker to approximately 60°C and titrate the excess oxalic acid with standard potassium permanganate solution.

A-4. CALCULATION

A-4.1 Aluminium phosphide (AlP) content, percent by mass

$$= \frac{0{\cdot}724\,7\,[\,(\,100 + A\,)\,N_1 - 100\,N_2\,]}{M}$$

where

A = volume, in ml, of the standard potassium permanganate solution required for the titration of excess oxalic acid;

N_1 = normality of standard potassium permanganate solution;

N_2 = normality of standard oxalic acid solution; and

M = mass, in g, of the material taken for the test.

APPENDIX B

(*Clause* 2.3.2)

DETERMINATION OF EVOLUTION OF PHOSPHINE GAS

[Caution — Phosphine gas is highly poisonous. Test shall be performed in a fume cupboard having a protective shield.]

B-1. APPARATUS

B-1.1 Humidified Chamber — consisting of a glass desiccator of diameter not less than 30 cm saturated with water vapours at room temperature.

B-2. PROCEDURE

B-2.1 Unpack three tablets/pellets directly from the original container on to a watch glass kept in the desiccator as quickly as possible. Keep the desiccator undisturbed for 30 minutes and observe the shape of tablets/pellets for any disintegration (losing their original shape) during this period. For comparison purposes a control be kept in a desiccator having an efficient and suitable dessicant like silica gel.

Caution — The tablets/pellets shall be removed from the desiccator as soon as the test is over.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	$1\ \mathrm{N} = 1\ \mathrm{kg\ m/s^2}$
Energy	joule	J	$1\ \mathrm{J} = 1\ \mathrm{N.m}$
Power	watt	W	$1\ \mathrm{W} = 1\ \mathrm{J/s}$
Flux	weber	Wb	$1\ \mathrm{Wb} = 1\ \mathrm{V.s}$
Flux density	tesla	T	$1\ \mathrm{T} = 1\ \mathrm{Wb/m^2}$
Frequency	hertz	Hz	$1\ \mathrm{Hz} = 1\ \mathrm{c/s}\ (\mathrm{s^{-1}})$
Electric conductance	siemens	S	$1\ \mathrm{S} = 1\ \mathrm{A/V}$
Electromotive force	volt	V	$1\ \mathrm{V} = 1\ \mathrm{W/A}$
Pressure, stress	pascal	Pa	$1\ \mathrm{Pa} = 1\ \mathrm{N/m^2}$

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